

# TEM Analysis of Diffusion-Bonded Silicon Carbide Ceramics Joined Using Metallic Interlayers

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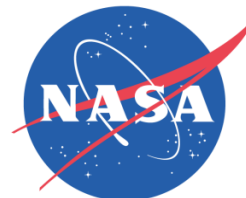
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## 1. Introduction

properties and applications of SiC (SA-THX)

purpose of diffusion bonding

## 2. Sample preparations used for diffusion bonding

Substrates : SA-Tyrannohex<sup>TM</sup> (SA-THX)

Interlayers : Ti-Mo, Ti-Cu foil

## 3. Experimental results

STEM images of the bonding area

TEM images and SAED patterns of the reaction compound

## 4. Discussion about the microstructure of the formed phases by diffusion bonding

## 5. Summary

# SiC fiber-bonded ceramics, SA-Tyrannohex ®

## SiC composite material

1. Excellent mechanical properties
2. Good oxidation resistance
3. High thermal stability

Especially,

### SA-Tyrannohex (SA-THX)

... SiC fiber-bonded ceramics

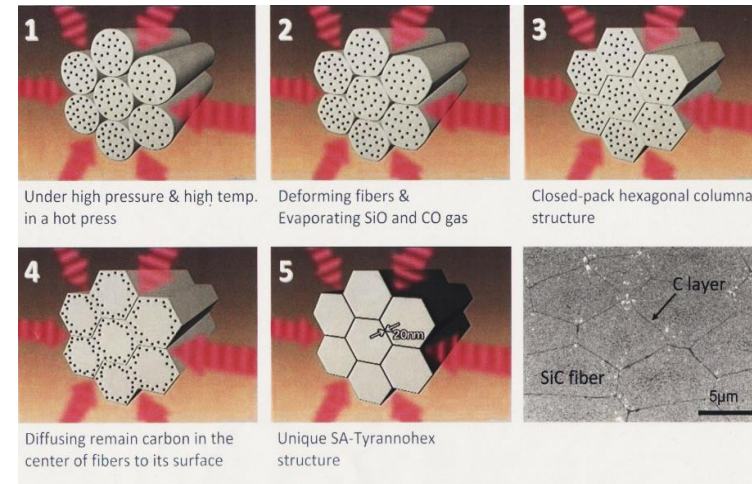
- High strength sustained **up to 1600°C in air**
- **High fracture toughness ( $1200 \text{ J} \cdot \text{m}^{-2}$ )**

⇒ Promising material for high-temperature  
and extreme environment applications

e.g. injector applications, combustion liner,  
nuclear fusion reactor and turbine engine applications



[http://www.ube-ind.co.jp/japanese/products/chemical/chemical\\_19.htm](http://www.ube-ind.co.jp/japanese/products/chemical/chemical_19.htm)



T. Ishikawa et al, *Science*, 282, 1295-1297 (1998).  
T. Ishikawa et al, *Nature*, 391, 773-775 (1998).

# For wide range uses of SA-THX

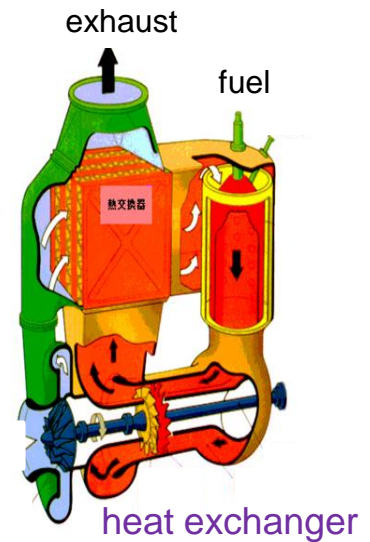
However, geometrical limitations hinder the wide use of SA-THX. It is difficult to fabricate large, or complex shaped components by Hot Pressing or CVD.

**Therefore, new advanced methods are needed.**

Under those circumstances,

One cost-effective solution for fabricating large, complex-shaped components is the **joining** of simple shaped ceramics.

**In this study, we are going to focus on diffusion bonding.**



# Diffusion Bonding of SA-THX using metallic Interlayers

## Used sample

@NASA

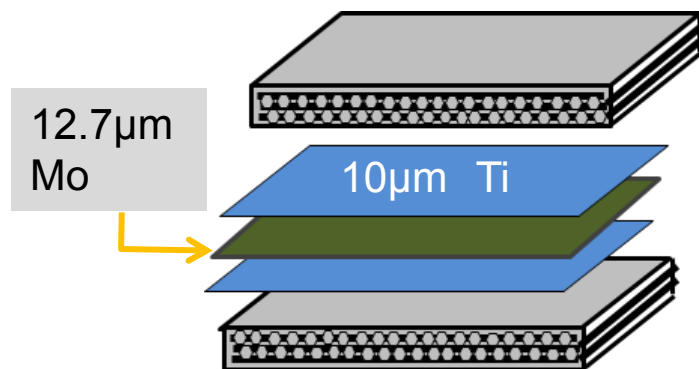
substrate: SA-THX ...SiC fiber-bonded ceramics, UBE Industries

metallic interlayer: Ti-foil, Mo-foil and Cu-foil, Goodfellow Corporation

## Bonding process

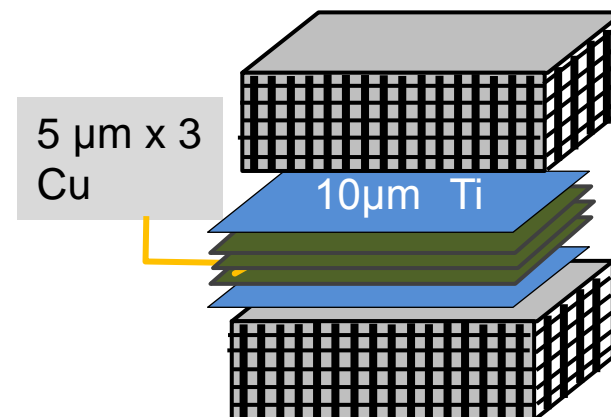
Hot-press in 1200°C, 4hour, vacuum 30MPa

Ti-Mo foil



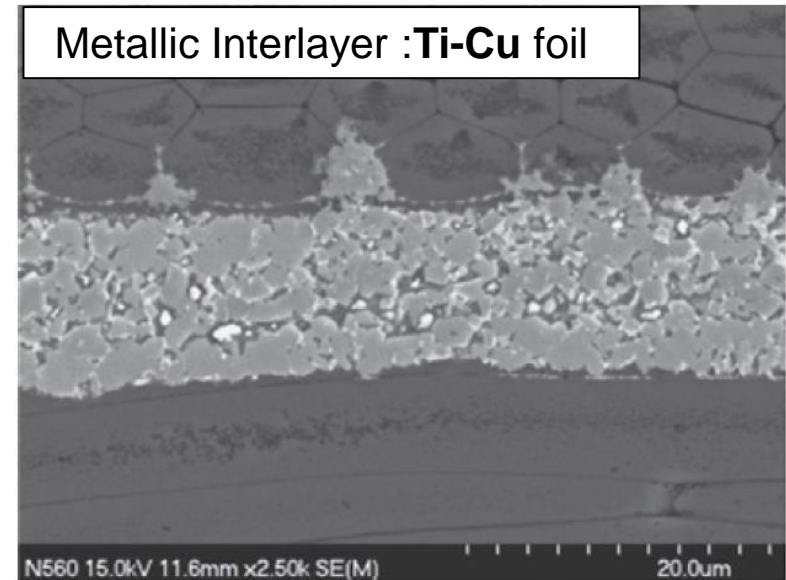
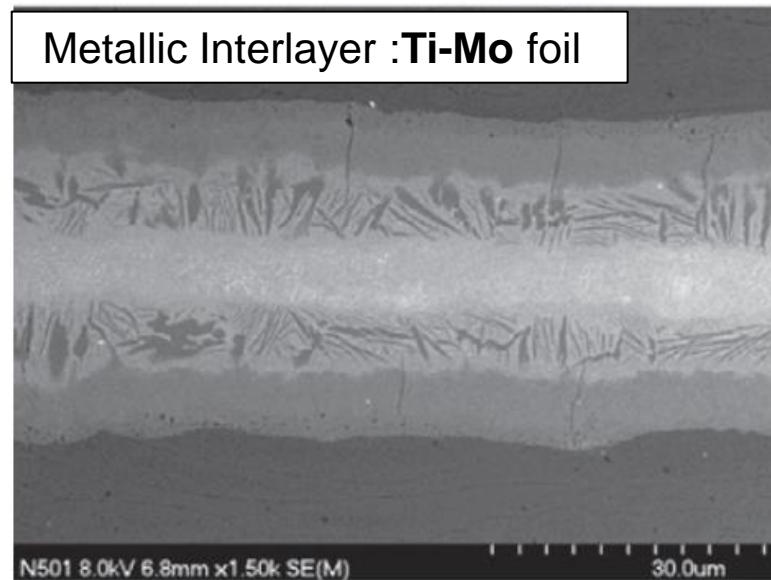
10µm Ti and 12.7 µm Mo interlayer

Ti-Cu foil



10µm Ti and 15µm Cu interlayer

# Diffusion Bonding of a SA-THX using metallic Interlayers



Knoop hardness of diffusion bonded joints.

	Average HK ( $\parallel$ joint)	Average HK ( $\perp$ joint)
SA-THX/Ti/Mo/Ti/SA-THX	<u><math>717.7 \pm 273.6</math></u>	$758.9 \pm 299.3$
SA-THX/Ti/Cu/Ti/SA-THX	<u><math>816.5 \pm 43.9</math></u>	—
SA-THX (un-bonded)	$1244 \pm 176$	$624 \pm 205$

M.C. Halbig, et. al., Ceramics International 41(2015)2140–2149

# Objectives

We diffusion bonded SiC and SiC (SA-THX and SA-THX) using Ti/Cu foil metallic interlayer.

We carried out TEM and STEM observations with the diffusion bonded sample prepared by FIB technique.

1. Evaluate microstructures of the diffusion bonding area by TEM and STEM.
2. Characterize the reaction compound in the diffusion bonding area by STEM-EDS and SAED analysis.

# Experiment ( FIB and STEM )

**Focused Ion Beam, FIB**  
(Hitachi FB-2200)



**Cs-corrected STEM**  
(Hitachi HD-2700)

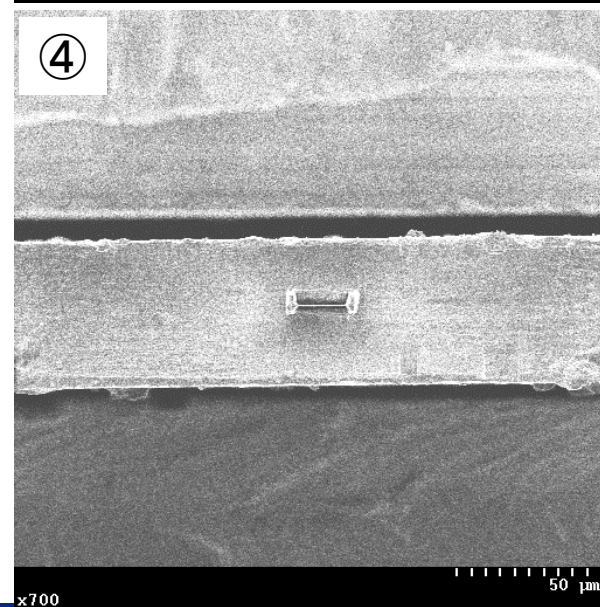
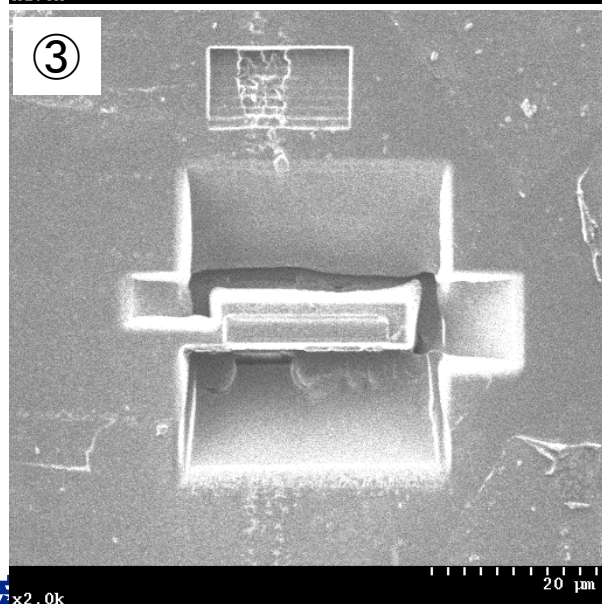
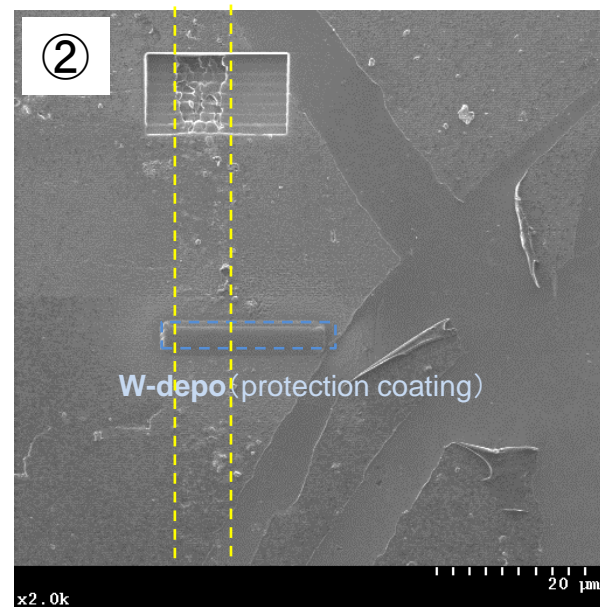
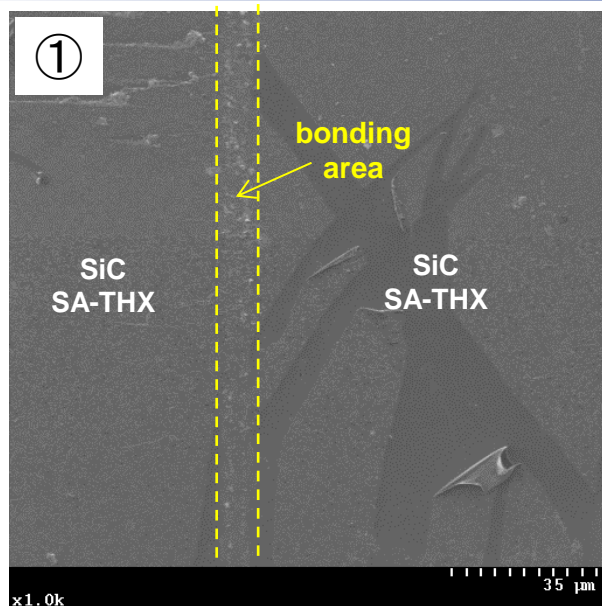


Prepared thin samples for TEM and STEM.

Checked the thin samples prepared by FIB.  
Three-Observation mode:

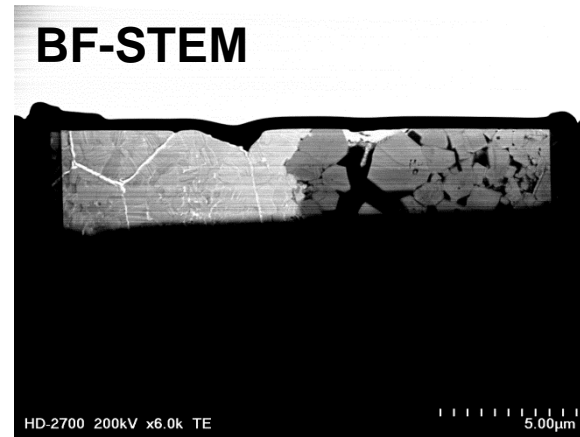
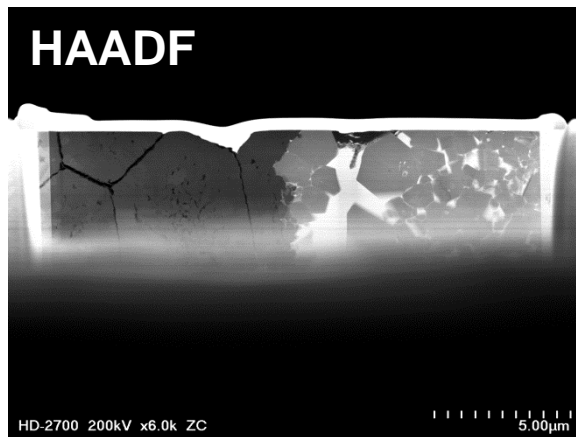
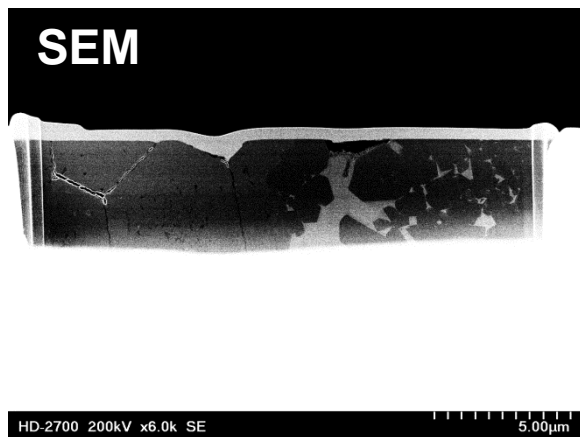
**SEM, BF-STEM and HAADF**

# Fabricating procedure of the thin sample (SIM image obtained by FIB)

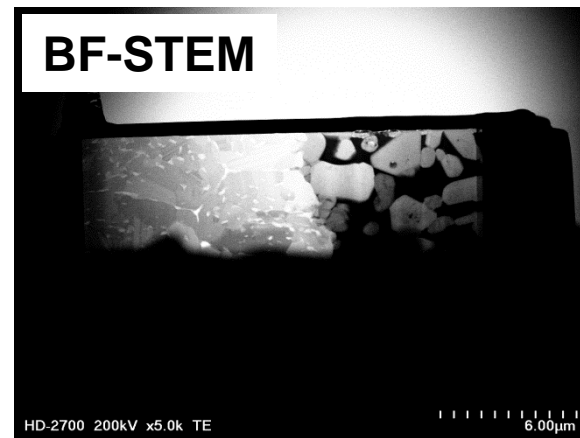
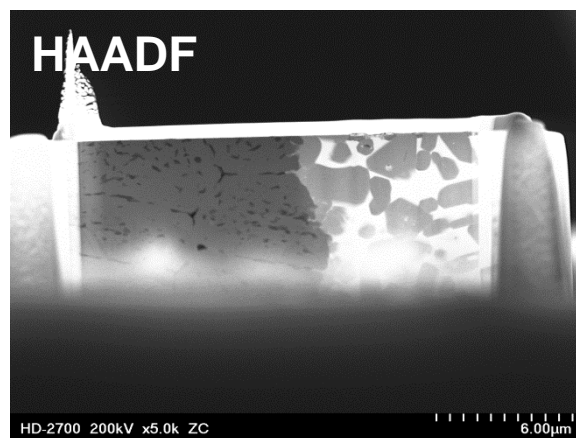
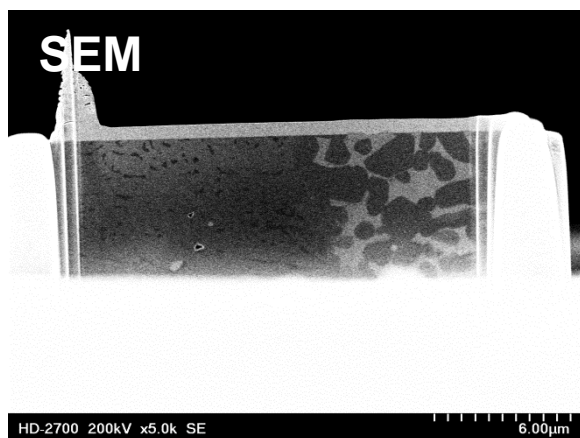


# STEM observation of the FIB sample

Ti/Cu interlayer **parallel** to SiC fiber



Ti/Cu interlayer **Perpendicular** to SiC fiber



HAADF

SiC  
(SA-THX)

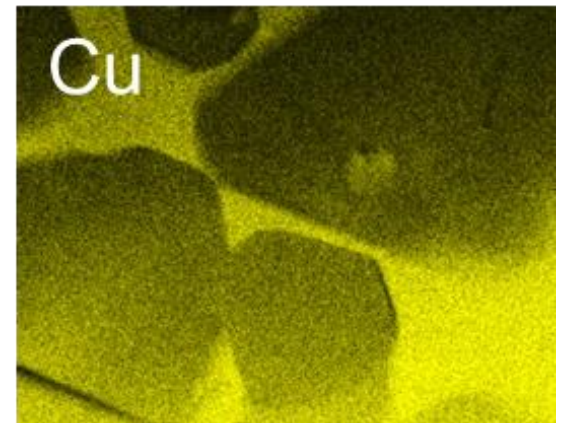
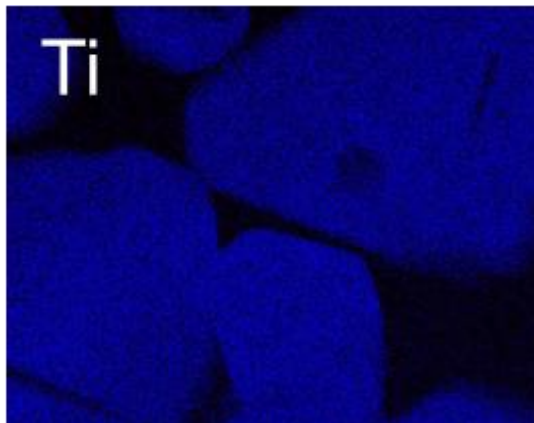
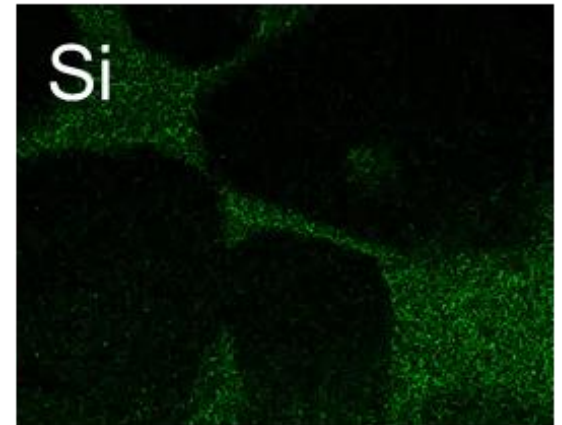
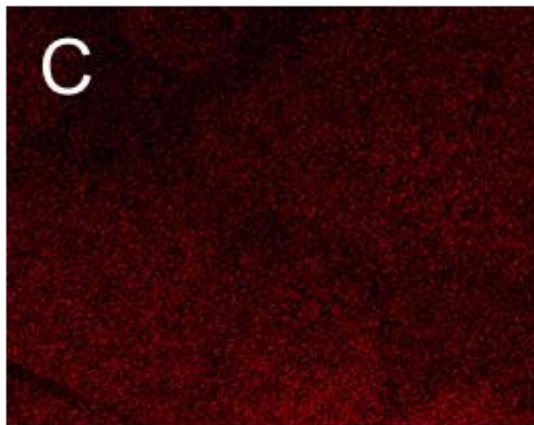
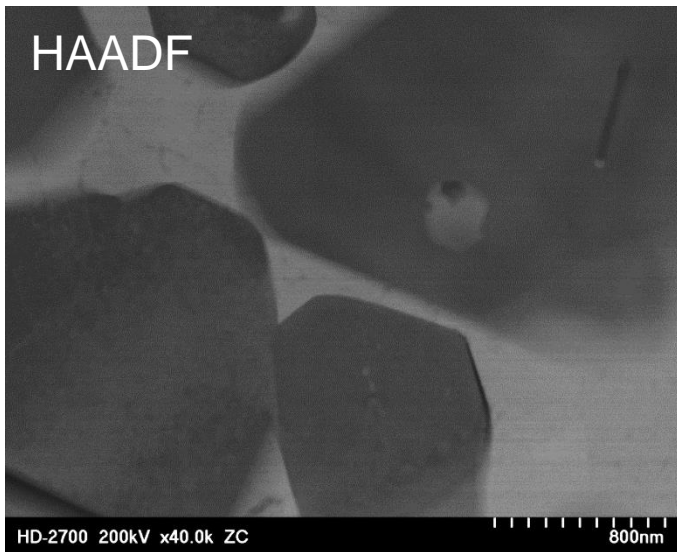
reaction  
layer

- Only one layer in the bonding area
- No diffusion toward the side of SiC
- The reaction layer is composed of some grains

and matrix around the grains.



# Element Mapping obtained by STEM-EDS analysis

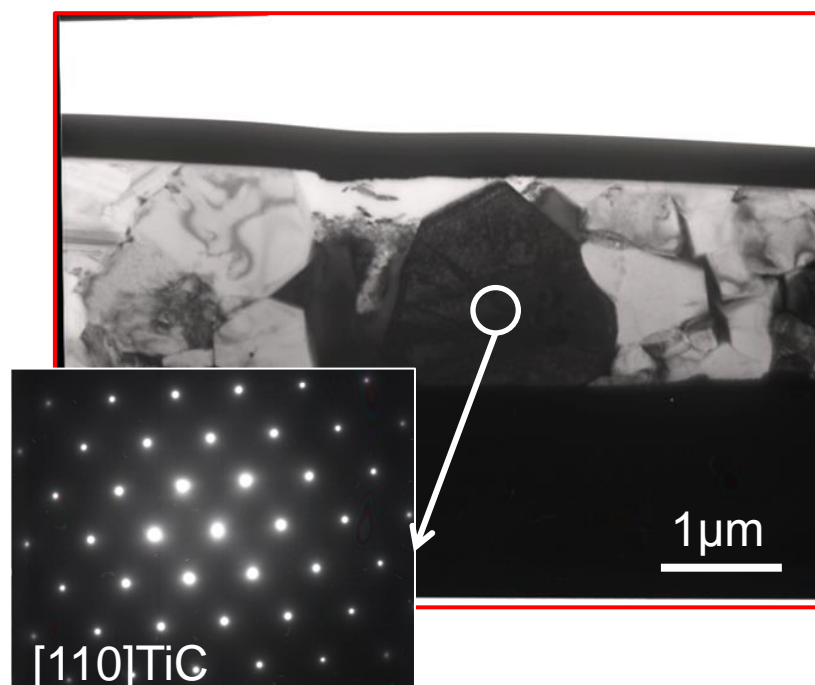
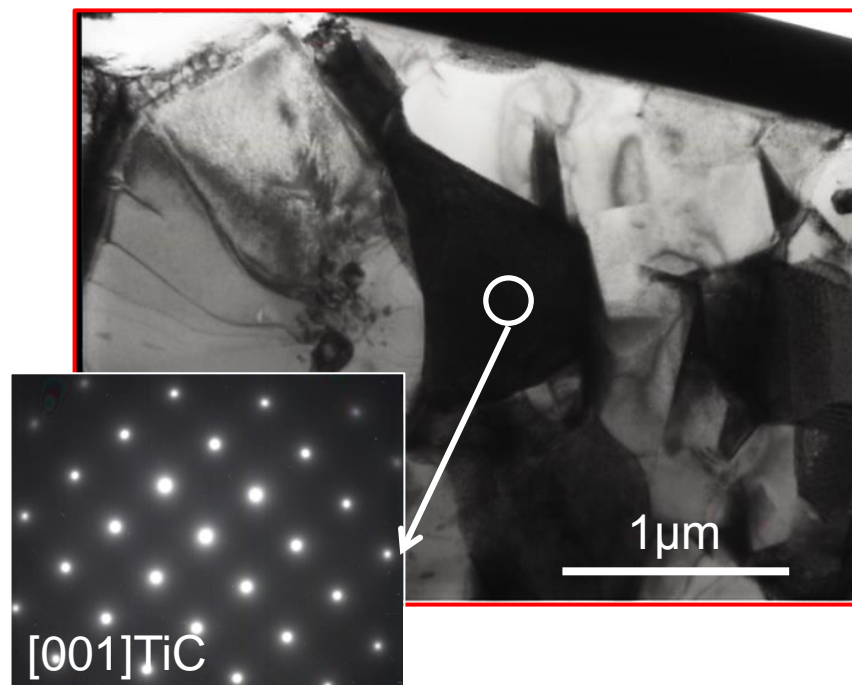


\* FIB mesh: Cu metal

Cu-Si matrix + precipitated TiC grains?



# TEM image and SAD patterns of Ti-C compound



**TiC**

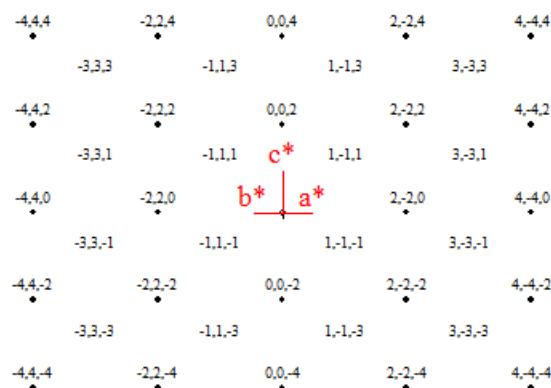
S.G.: Fm3m

NaCl-type

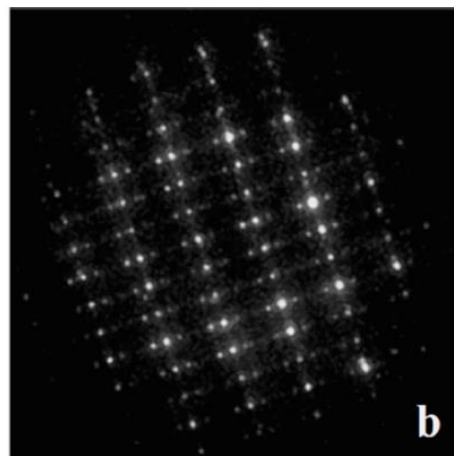
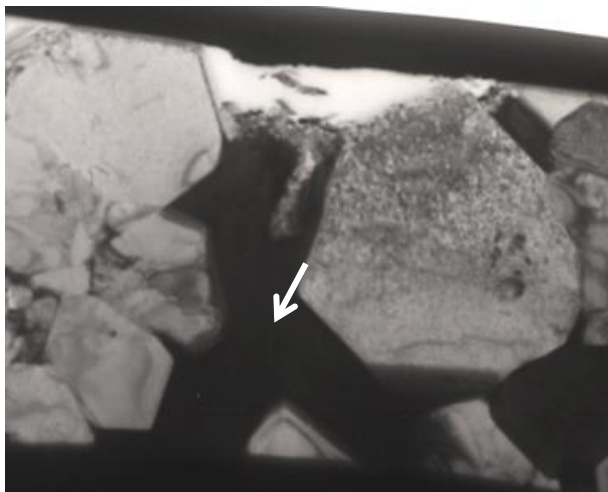
[001]TiC



[110]TiC



# TEM image and SAD patterns of Cu-Si compound

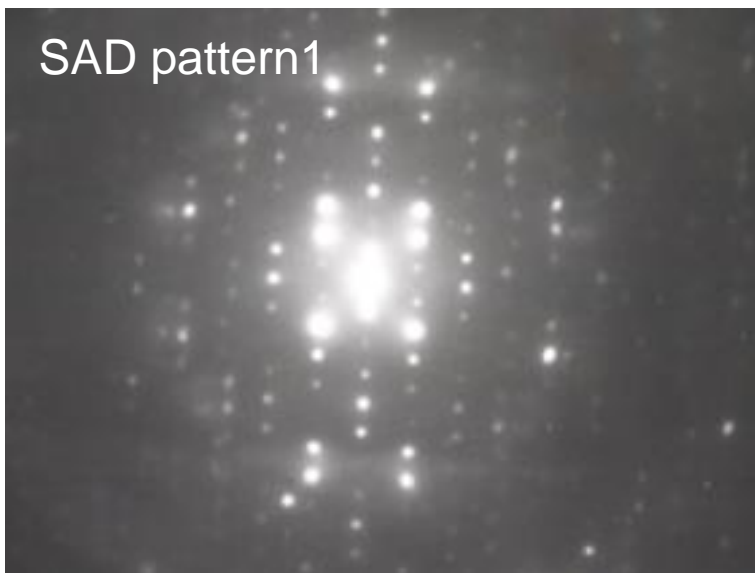


SAD pattern of  $\text{Cu}_3\text{Si}$   
:  $\eta''$ -phase  
(RT phase of  $\text{Cu}_3\text{Si}$ )

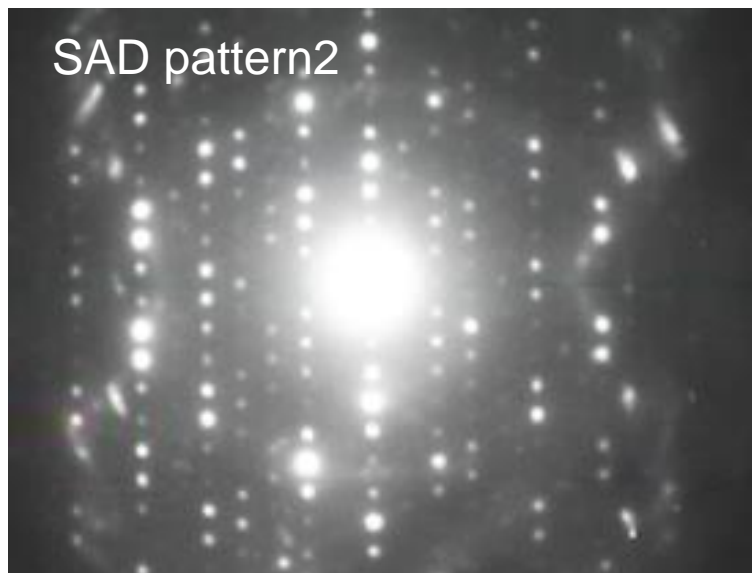
//[001]  
4 times superstructure  
//[111]  
3 times superstructure

M. Heuer, *et. al.*, JAP 101, 123510 (2007)

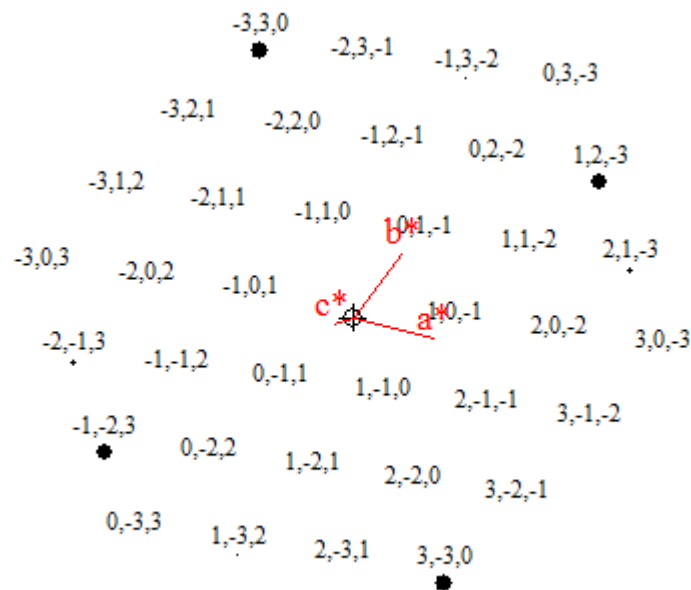
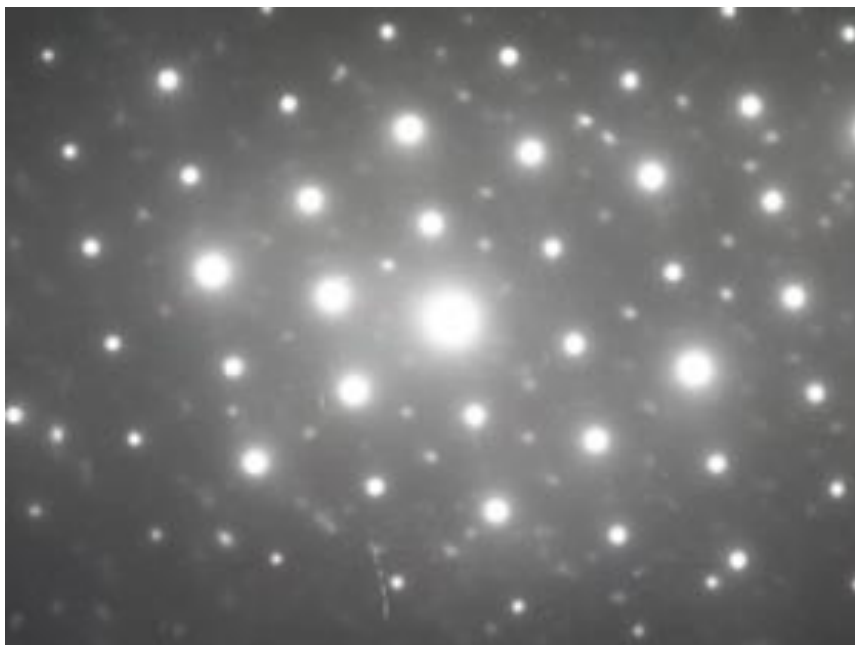
SAD pattern1



SAD pattern2



# TEM image and SAD patterns of Cu-Si compound



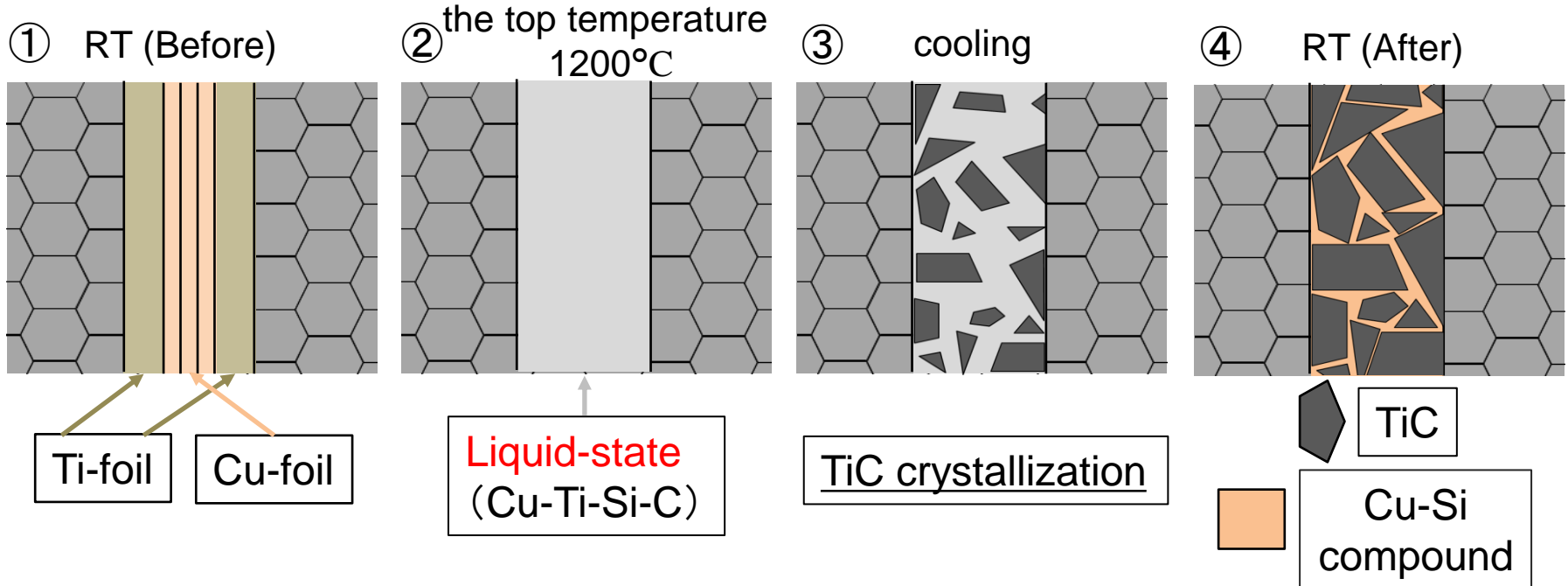
$\eta$ -phase  $\text{Cu}_3\text{Si}$ :

(high temperature phase)

S.G.: P-3m1

$a=0.4091$  nm,  $b=0.7358$

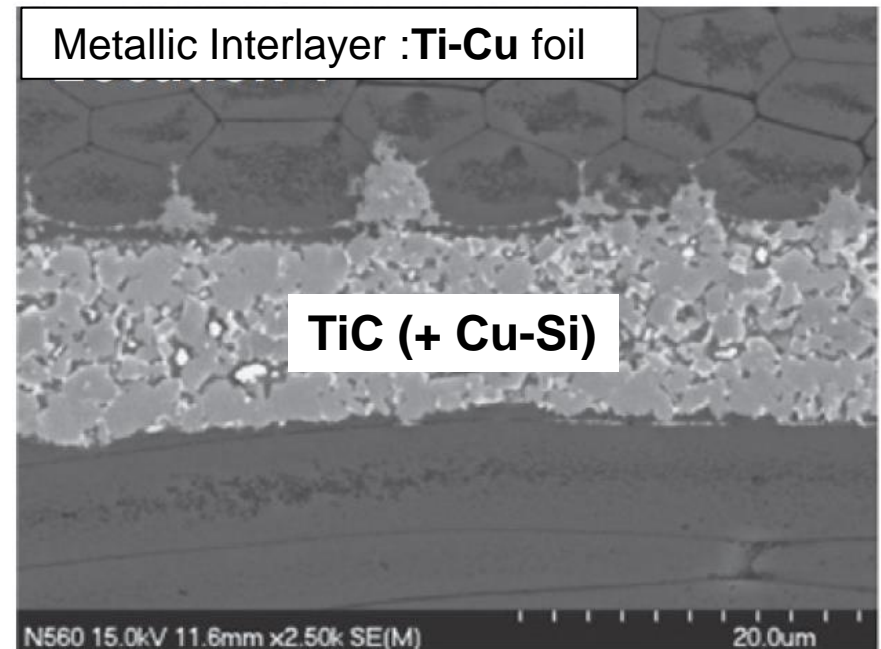
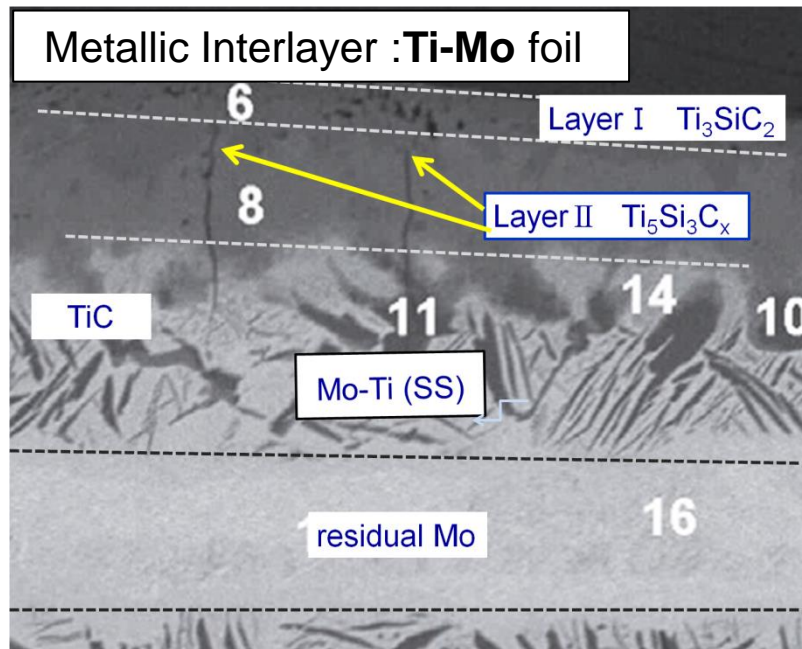
# Consideration of the Results



The bonding layer is composed of TiC precipitations in Cu-Si matrix.

Cu-Si matrix plays a role of the binder of TiC precipitations?

# Consideration of the Results (CTE)



coefficient of thermal expansion (CTE;  $\alpha$ )

material	a-SiC	Mo	Ti	Cu	TiC	Ti <sub>3</sub> Si <sub>5</sub>		Ti <sub>3</sub> SiC <sub>2</sub>	
						a	c	a	c
CTE $\alpha$ ( $10^{-6}\text{K}^{-1}$ )	3.2	5.1	8.4	16.8	7.4	6.1	16.6	8.9	10

M.C. Halbig, et. al., Ceramics International 41(2015)2140–2149

## Summary

- 1. We picked up thin samples from the bonded area of diffusion bonded SA-THX by a FIB micro-sampling technique. The prepared thin samples were sufficiently thin and less-damaged, and allowed the detailed evaluation by TEM and STEM.
- 2. The microstructure of diffusion bonded area was observed by STEM and TEM. The composition and crystal structures of the reaction compound were investigated by STEM-EDS and SAED method. The reaction layer of the diffusion bonding was composed of TiC precipitations in Cu-Si compound matrix.

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